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FY93 End of Fiscal Year Letter
(01 Oct 1992 - 30 Sep 1993)

ONR CONTRACT INFORMATION

Contract Title: Micromechanics of Interfaces in Metal Matrix Composites

Performing Organization: Chemical Engineering & Materials Science, University of Minnesota, Minneapolis, MN 55455

Principal Investigator: William W. Gerberich

Contract Number: N00014-92-J-1962

R & T Project Number: 4313299---01

ONR Scientific Officer: Dr. Steven Fishman

Enclosure (1)

A. Scientific Research Goals

Defining the role of the interface is critical to understanding mechanical properties of metal matrix composite materials. Measurements of interfacial fracture toughness are being conducted to aid in the development of MMC's. The scientific research goals are to develop a better understanding of how nonlinear behavior in the metal matrix and chemical bonding or frictional effects at the bi-material interface combine to provide the interfacial fracture toughness. To accomplish that a number of spectroscopies including RBS and several fracture mechanics approaches including fiber push-out are being employed. These will provide feedback to current processing conditions utilized in MMC's. The practical application is to assist 3M in the Model Factory program.

B. Significant Results

Initial studies of flat plate configurations using both HIPed processing of UCSB specimen configurations and thermally reacted thin films have demonstrated how metal film thickness dissipates plastic energy. In evaluations which covered a three order of magnitude variation in thickness, the interfacial fracture toughness dropped from about 3 MPa-m$^{1/2}$ to 0.2 MPa-m$^{1/2}$. This latter value is consistent with the true work of adhesion of the system. In other work, we had already shown that unreacted and reacted systems have a large bearing on the fracture toughness of thin film systems. The implication is that the *true work of adhesion* plays not only a specific role but a *significant role* in how much energy is dissipated in the practical work of adhesion. As such, thin film microscratch studies on reacted interfaces with specially prepared surfaces or chemistries should be able to project how that interface will change the fiber/matrix interface performance and thus the overall performance of the composite as it depends on the mode II fracture toughness.

For example, we have been able to measure the mode II interfacial fracture energy from fiber push out tests. In the Al_2O_3 fiber, β-21STi composite system, for the first time to our knowledge, we have used a nanoindentation procedure to *push out 10 μm diameter fibers.* Using Liang and Hutchinson's analysis and the load drop, ΔP, during fiber push out, one can calculate Γ_{II}. This is obtained from

$$\Delta P = \pi R^2 \left[\sigma_f + 2\sqrt{\frac{\Gamma_{II} E_f}{B_2 R}} \exp\left(\frac{2B_1 \mu t}{R} \right) \right]$$

where B_1, B_2 are material constants, R is the fiber radius, σ_f is the axial residual stress, Γ_{II} is the mode II interfacial fracture energy, μ is the interfacial friction coefficient, and t is the thickness of the material the fiber is being pushed through. From a fiber sliding model, we were able to match up the interfacial frictional stress of about 310 MPa and the fiber displacement as it was being pushed through. This resulted in a friction coefficient of about 0.32. With this and the measured load drop, the above gives Γ_{II} to be 0.13 J/m^2. We have compared this to a lower bound estimate for thermally reacted Al_2O_3/Ti interfaces, which gives a true work of adhesion of 0.18 J/m^2. This demonstrates that the diffusion barrier used in the composite system reduced the interfacial reactions such that there was negligible plasticity in the surrounding matrix during pushout. This can be partially understood from the relative smoothness of the fibers which are 18.8 ± 0.2 nm as measured by Atomic Force Microscopy. It is also reasonably consistent with an interfacial frictional stress of 312 ± 2 MPa which is substantially below the flow stress of the

321STi matrix. While the 0.13 J/m^2 energy is between a refractory metal barrier and Y_2O_3, it is nevertheless the nonlinear behavior in the matrix that would contribute mostly to increases in toughness. The next step is to prove these techniques — the measurement of fracture toughness by microscratch of thin film analogies, the complementary analysis provided by fiber push-out studies and AFM analysis of surface roughness and friction coefficients — on an interfacial system that is step-wise varied in terms of the processing conditions.

C. Future Work

Expectations are that such approaches will eventually be applicable to measuring the effects of surface roughness and chemistry changes on the performance of metal matrix composites. We have been discussing this with 3M personnel and it would appear that SiC fibers are now favored. What would be done is to prepare a combination of flat plate analogies of SiC whiskers with various surface modifications and deposit thin films of aluminum or titanium alloys by evaporation or sputtering. Additional processing would involve thermal mechanical step(s). Direct comparisons of these to MMC's with SiC fibers subjected to similar surface modifications would be attempted. A consistency would be sought regarding how well fracture micromechanics can be used to predict the variations in interfacial fracture toughness with processing as observed from the two approaches.

D. List of Publications/Reports/Presentations

1. Papers Published in Refereed Journals

H.F. Wang, W.W. Gerberich and C.J. Skowronek, "Fracture Mechanics of Ti/Al_2O_3 Interfaces," *Acta Metall. Mater.* **41** (1993) pp. 2425–2432.

H.F. Wang and W.W. Gerberich, "Effect of a Diffusion Barrier on The Interfacial Chemistry and Mechanical Properties of Ti/Al_2O_3 Composites," submitted, *Metall. Trans. A*, June (1993).

H.F. Wang, J.C. Nelson, W.W. Gerberich and H.E. Deve, "Evaluation of In-Situ Mechanical Properties of Composites by using Nanoindentation Techniques," in press, *Acta Metall. Mater.* (1993).

Y.-C. Lu, S.L. Sass, Q. Bai, D.L. Kohlstedt and W.W. Gerberich, "The Influence of Interfacial Reactions on The Mechanical Properties of Ti-Al_2O_3 Interfaces," submitted, *Acta Metall. Mater.*, July 1993.

H.F. Wang, J.C. Nelson, C.-L. Lin and W.W. Gerberich, "Interfacial Stability and Mechanical Properties of Al_2O_3 Fiber Reinforced Ti Matrix Composites," submitted, *J. Mat. Res. Soc.*, July 1993.

H.F. Wang, S. Venkataraman, W.W. Gerberich, Q. Bai, T. Wu and D.L. Kohlstedt, "Metal Thickness and Processing Temperature Effects on Ti/Al_2O_3 Interface Fracture," submitted, *J. Mater. Res. Soc.*, Feb. 1993.

2. Non-Refereed Publications and Published Technical Reports

(none)

3. Presentations

   a. Invited

   b. Contributed

   With S.K. Venkataraman, H.-F. Wang, C.-L. Lin and D.L. Kohlstedt, "Macro- and Micro-Measures of Film Thickness Contributions to Metal-Ceramic Interface Toughness," Symposium 1, MRS, Boston, November 30, 1992.

   With H.F. Wang, C.J. Skowronek and H.M. Meyer, "Reaction Zone Effects on the Interfacial Fracture Toughness of Metal Matrix Composites," TMS Fall Meeting, Chicago, November 2, 1992.

   With H.F. Wang, J.C. Nelson, C.J. Skowronek and H.E. Deve, "Evaluation of Adhesion Strength in Metal/Ceramic Composites," Spring MRS, Symposium R, San Francisco, April 12, 1993.

4. Books (and sections thereof)

   H.F. Wang, J.C. Nelson, C.L. Lin, W.W. Gerberich, C.J. Skowronek and H.E. Deve, "Evaluation of Adhesion Strength in a Ti/Al_2O_3 Composite," accepted, *Mater. Res. Symp. R.* Spring (1993).

E. List of Honors/Awards

William W. Gerberich	Univ. of Minnesota	Plenary Lecturer (1 of 4) for Corrosion/ Deformation Interaction '92, sponsored by Electricitie de France, CNRS FRAMATOME; selected to be on '96 Organizing Committee.
William W. Gerberich	Univ. of Minnesota	Elected Vice-Chairman of the Board of Governors for the NSF Sponsored Institute for Mechanics and Materials. The purpose of the board is to be an oversight body for the Institution's educational mission of fostering interdisciplinary understanding between the mechanics and materials communities.

F. Participants (1992–93)

1. Professor William W. Gerberich, Chemical Engineering and Materials Science has oversight responsibility for Mr. Hsin-fu Wang, Mr. Michael Kriese, graduate students and Dr. John Nelson, Post-Doctoral Associate.

2. Professor David A. Kohlstedt, Geology and Geophysics has oversight responsibility for Drs. Quan Bai and Brad Benson, Post-doctoral Associates.

3. Mr. Hsin-fu Wang has had major responsibility for performing characterization studies and coordinating processing assistance from 3M personnel. He will be finishing this year.

4. Mr. Michael Kriese is a new graduate student who will assume Mr. Hsin-fu Wang's responsibilities.

5. Dr. John Nelson is in charge of the continuous microindentation facility and has assisted in fiber push-out studies.

6. Drs. Quan Bai and Brad Benson have assisted with high temperature reaction studies and thin-film mechanics.

7. Dr. H. Deve and Mr. C. Skowronek from 3M have assisted with interfacial fracture mechanics and composite processing.

G. Other Sponsored Research (1992-93)

1. DOE, Basic Energy Sciences Grant DE-FG02-84ER45141, "Micromechanisms of Brittle Fracture: STM, TEM and Electron Channeling Analysis," $84,584, 7/1/92–6/30/93, 1/2 month summer salary.

2. DOE, Basic Energy Sciences Grant DE-FG02-88ER45337, "A Study of Scale Cracking and its Effect on Oxidation and Hot Corrosion," $36,540, 11/1/92–10/31/93, 1/2 month summer salary.

3. NSF, Center for Interfacial Engineering (with 19 other P.I.'s), $2,600,000, 10/1/92–9/30/93, support for 1 1/2 graduate students, 1/2 post-doctoral associate, no summer salary.

4. ONR Grant N/N00014-89-1726, "Microstructure and Texture Control of Fatigue in Hydrogen and Marine Environments," $86,363, 3/1/92–2/28/93, 1/2 month summer support.

5. Cardiac Pacemakers Inc., "Corrosion Studies of Biomedical Materials," $42,000, 1/30/93–2/1/94, 1 month summer salary.

6. NSF, Center for Plasma Aided Manufacturing, 10/1/92–9/30/93, support for 2 co-advised students (Professors Pfender and Heberlein, Mechanical Engineering) no summer salary.

H. SUMMARY OF FY93
PUBLICATIONS/PATENTS/PRESENTATIONS/HONORS/PARTICIPANTS
(Number Only)

		ONR	non ONR
a.	Number of Papers Submitted to Referred Journal but not yet published:	5	12
b.	Number of Papers Published in Refereed Journals:	1	13
c.	Number of Books or Chapters Submitted but not yet Published:	1	11
d.	Number of Books or Chapters Published:	-	7
e.	Number of Printed Technical Reports & Non-Referred Papers:	-	-
f.	Number of Patents Filed:	-	-
g.	Number of Patents Granted:	-	1
h.	Number of Invited Presentations at Workshops or Prof. Society Meetings:	-	5
i.	Number of Contributed Presentations at Workshops or Prof. Society Meetings:	3	14
j.	Honors/Awards/Prizes for Contract/Grant Employees: (selected list attached)	-	2
k.	Number of Graduate Students and Post-Docs Supported at least 25% this year on contract grant:	2	11
	Grad Students: TOTAL	1	10
	Female	0	2
	Minority	0	0
	Post Doc: TOTAL	1	1
	Female	0	0
	Minority	0	0
l.	Number of Female or Minority PIs or CO-PIs		
	New Female	0	0
	Continuing Female	0	0
	New Minority	0	0
	Continuing Minority	0	0

Enclosure (4)